

## **EXHIBIT B**



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**Kealey et al.**

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 (45) **Date of Patent:** **\*Nov. 6, 2001**

(54) **COCOA COMPONENTS, EDIBLE PRODUCTS HAVING ENRICHED POLYPHENOL CONTENT, METHODS OF MAKING SAME AND MEDICAL USES**

(58) **Field of Search** ..... 426/631, 593,  
 426/542; 549/386; 424/195.1

(56) **References Cited**

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**U.S. PATENT DOCUMENTS**

2,417,078 3/1947 Jones ..... 241/48  
 2,558,854 7/1951 Kempf et al. .... 99/23  
 2,771,927 11/1956 Thaning ..... 146/227

(List continued on next page.)

**FOREIGN PATENT DOCUMENTS**

1379116 1/1975 (GB) ..... B02B/5/00  
 WO96/10404 4/1996 (WO) .  
 WO99/45788 \* 9/1999 (WO) .

**OTHER PUBLICATIONS**

Beckett, S.T., Ed., "Industrial Chocolate Manufacture and Use", 2<sup>nd</sup> Ed., Published by Blackie Academic Professional (an imprint of Chapman & Hall) Glasgow, U.K., 1994, by Chapman & Hall, pp. 55-82.

(List continued on next page.)

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This patent is subject to a terminal disclaimer.

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(52) **U.S. Cl.** ..... **426/631; 426/542; 426/593; 549/386; 424/195.1**

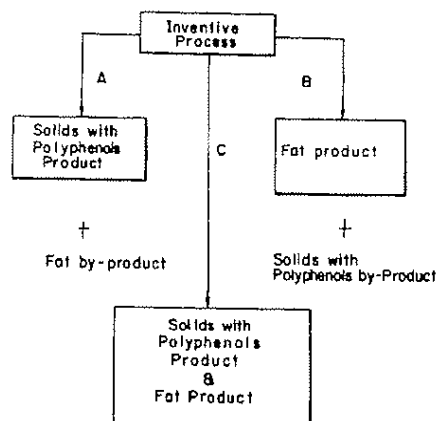
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(57) **ABSTRACT**

Cocoa components having enhanced levels of cocoa polyphenols, processes for producing the cocoa components while conserving a significant amount of the cocoa polyphenols, compositions containing the cocoa components or the cocoa polyphenols, and methods of using the cocoa components or the cocoa polyphenols for improving the health of a mammal are described. The cocoa components include partially and fully defatted cocoa solids, cocoa nibs and fractions derived therefrom, cocoa polyphenol extracts, cocoa butter, chocolate liquors, and mixtures thereof. The invention provides processes for extracting fat from cocoa beans and for otherwise processing cocoa beans to yield a cocoa component having conserved concentrations of polyphenols relative to the starting materials.

**33 Claims, 5 Drawing Sheets**



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**U.S. PATENT DOCUMENTS**

3,141,777	7/1964	Guidarelli et al. ....	99/88	5,505,982	4/1996	Krawczyk et al. ....	426/660
3,904,777	9/1975	Goerling et al. ....	426/631	5,554,645	* 9/1996	Romanczyk ....	514/453
3,923,847	12/1975	Roselius et al. ....	260/412.8	6,015,913	* 1/2000	Kealey et al. ....	549/386
3,955,489	5/1976	Goerling et al. ....	99/483	6,194,020	* 2/2001	Myers ....	426/631
3,997,680	12/1976	Chalin ....	426/262	<b>OTHER PUBLICATIONS</b>			
4,235,939	11/1980	Kimberley, Sr. ....	426/549	Minifie, Bernard W., <i>Chocolate, Cocoa and Confectionery: Science and Technology</i> , 3 <sup>rd</sup> Ed., Published by Chapman & Hall, Nw York, 1989, by Van Nostrand Reinhold, pp. 31-51 and 61-76.			
4,271,754	6/1981	Homann ....	100/37	Swern, D., Ed., <i>Bailey's Industrial Oil and Fat Products</i> , 4 <sup>th</sup> Ed., John Wiley & Sons, New York, NY, (1982) vol. 2. pp 175-251.			
4,322,444	3/1982	Zuilichem et al. ....	426/241	Wood, G.A.R., "Cocoa" 4 <sup>th</sup> Edition, Longman Scientific and Technical, Essex, England (1985), pp510-513.			
4,435,436	3/1984	Terink et al. ....	426/631	Kashket et al. <i>Arch. Oral Biol.</i> 30:11-12 821-6 (1985).			
4,444,798	4/1984	Magnolato et al. ....	426/422	Clapperton J., et al., "Polyphenols and Chocolate Flavour." Proc. Group Polyphenols, Lisbon. Portugal, (1992).			
4,701,337	10/1987	Frost et al. ....	426/660	Cook, L. Russell et al., "Chocolate Production and Use", Published by Harcourt Brace Jovanovich, Inc., New York, NY (1982), pp. 143-155 and 162-172.			
4,704,292	11/1987	Kattenberg ....	426/565	* cited by examiner			
4,758,444	7/1988	Terauchi et al. ....	426/593				
4,784,866	11/1988	Wissgott ....	426/262				
4,810,516	3/1989	Kong-Chan ....	426/548				
4,871,562	10/1989	Terauchi et al. ....	426/330.3				
4,999,197	3/1991	Wursch ....	424/195.1				
5,009,917	4/1991	Wiant et al. ....	426/631				
5,114,730	5/1992	Ellis ....	426/593				
5,244,099	9/1993	Zaltzman et al. ....	209/466				
5,252,349	10/1993	Carter, Jr. ....	426/482				
5,405,633	4/1995	Heidlas et al. ....	426/442				
5,464,649	11/1995	St. John et al. ....	426/660				
5,474,795	12/1995	Surber et al. ....	426/660				

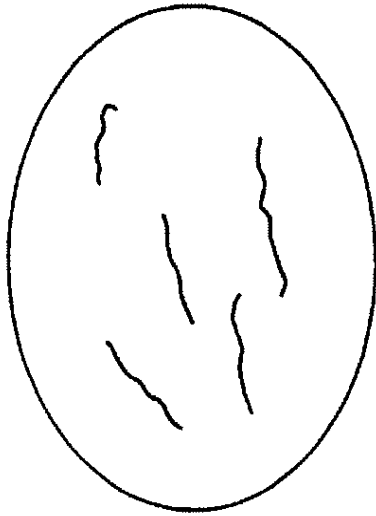
**U.S. Patent**

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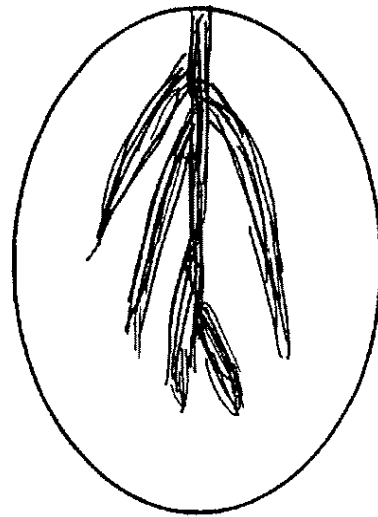
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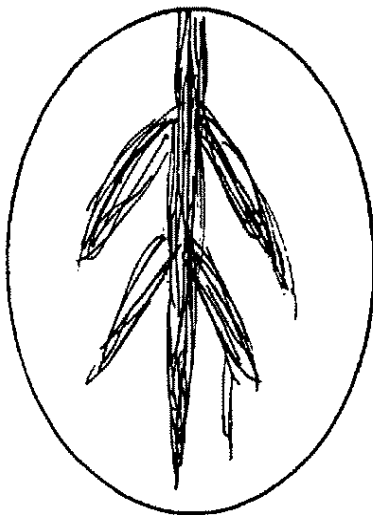
*FIG. 1a*



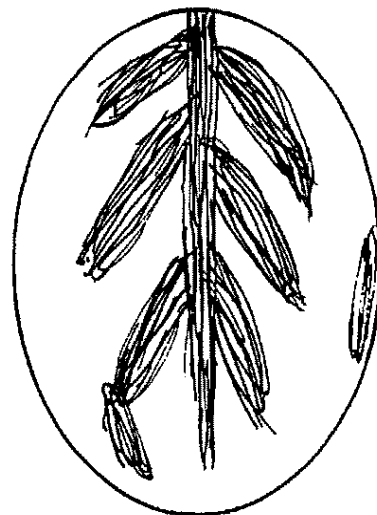
*FIG. 1b*



*FIG. 1c*



*FIG. 1d*



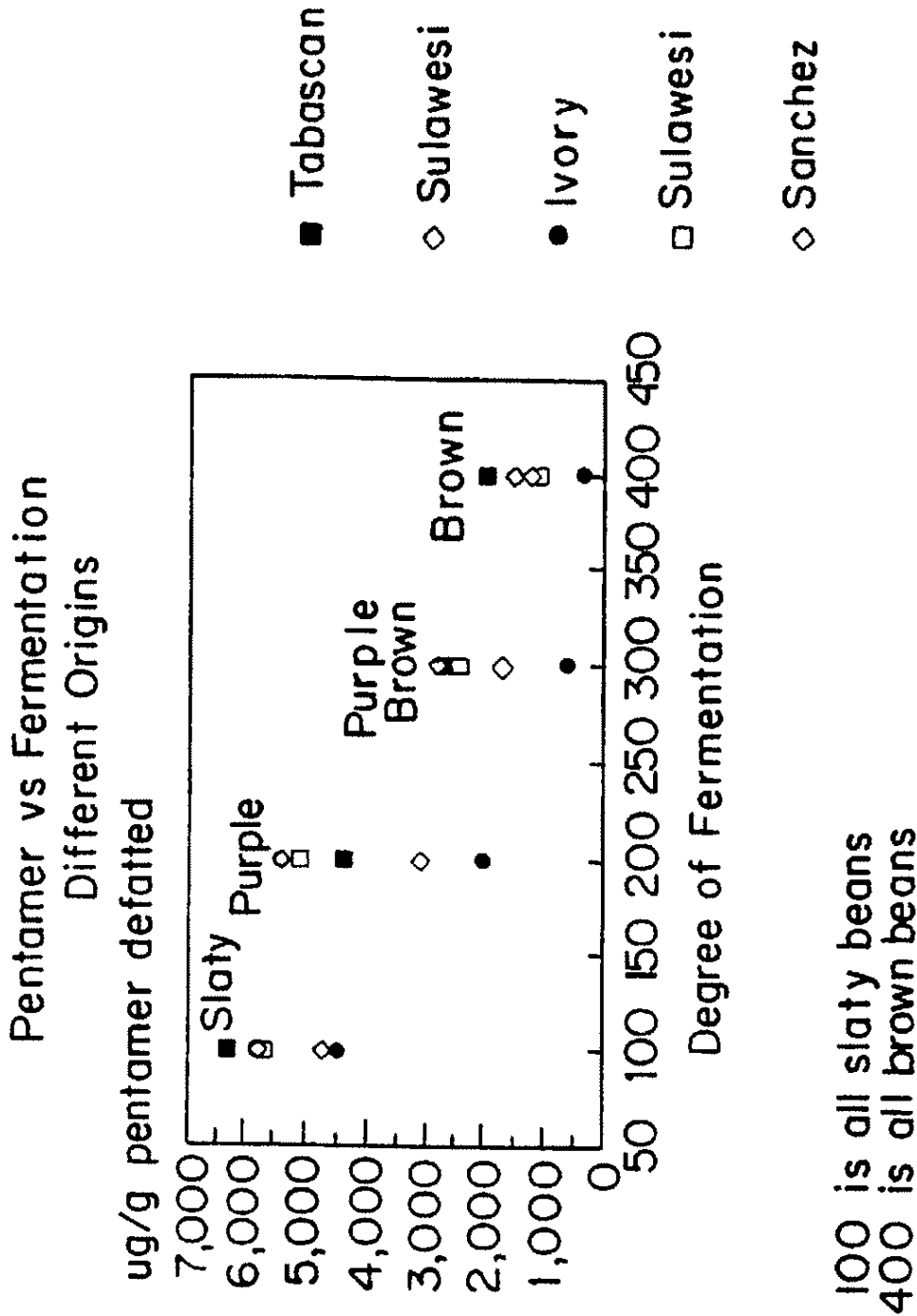


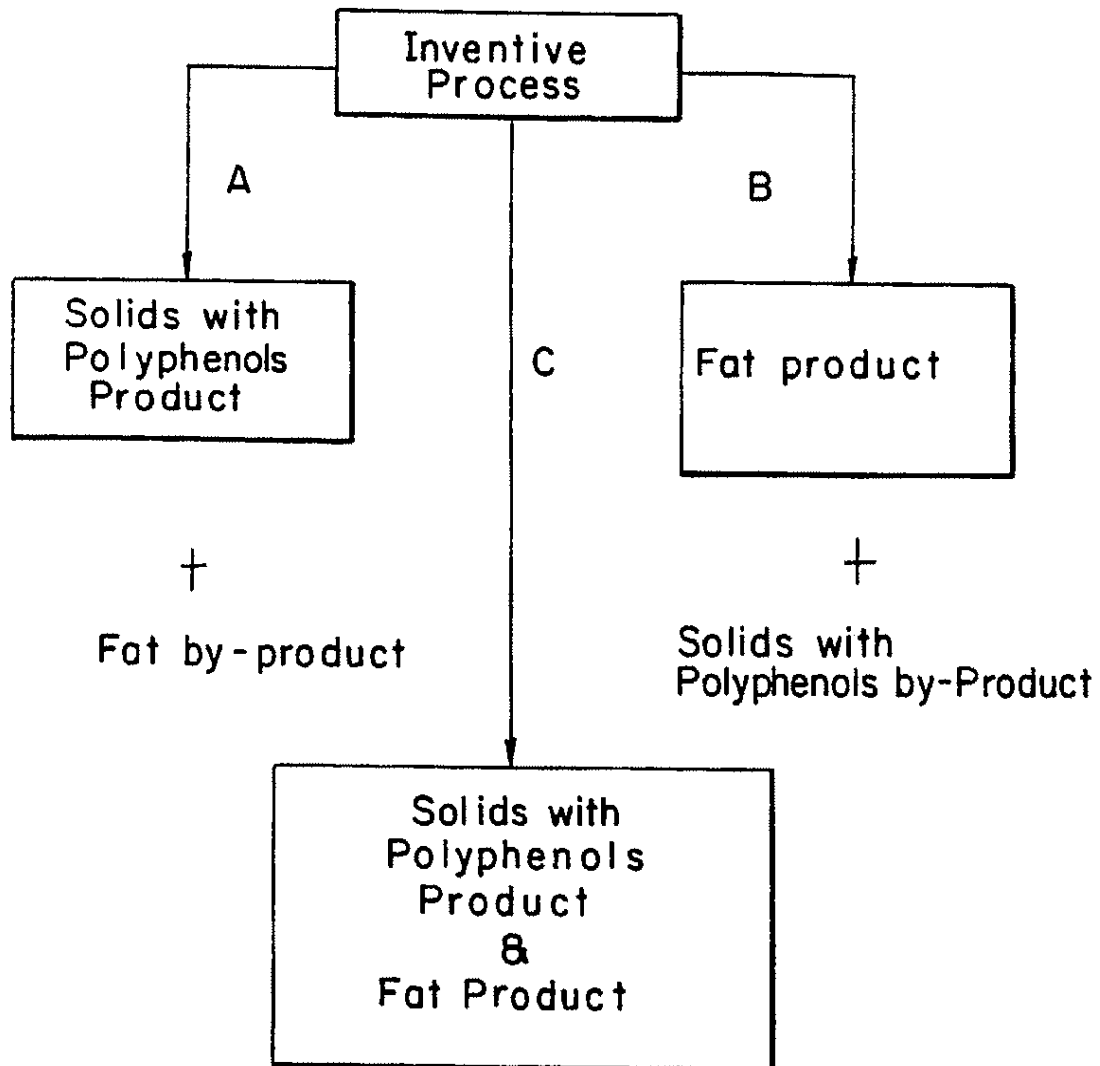
FIG. 2

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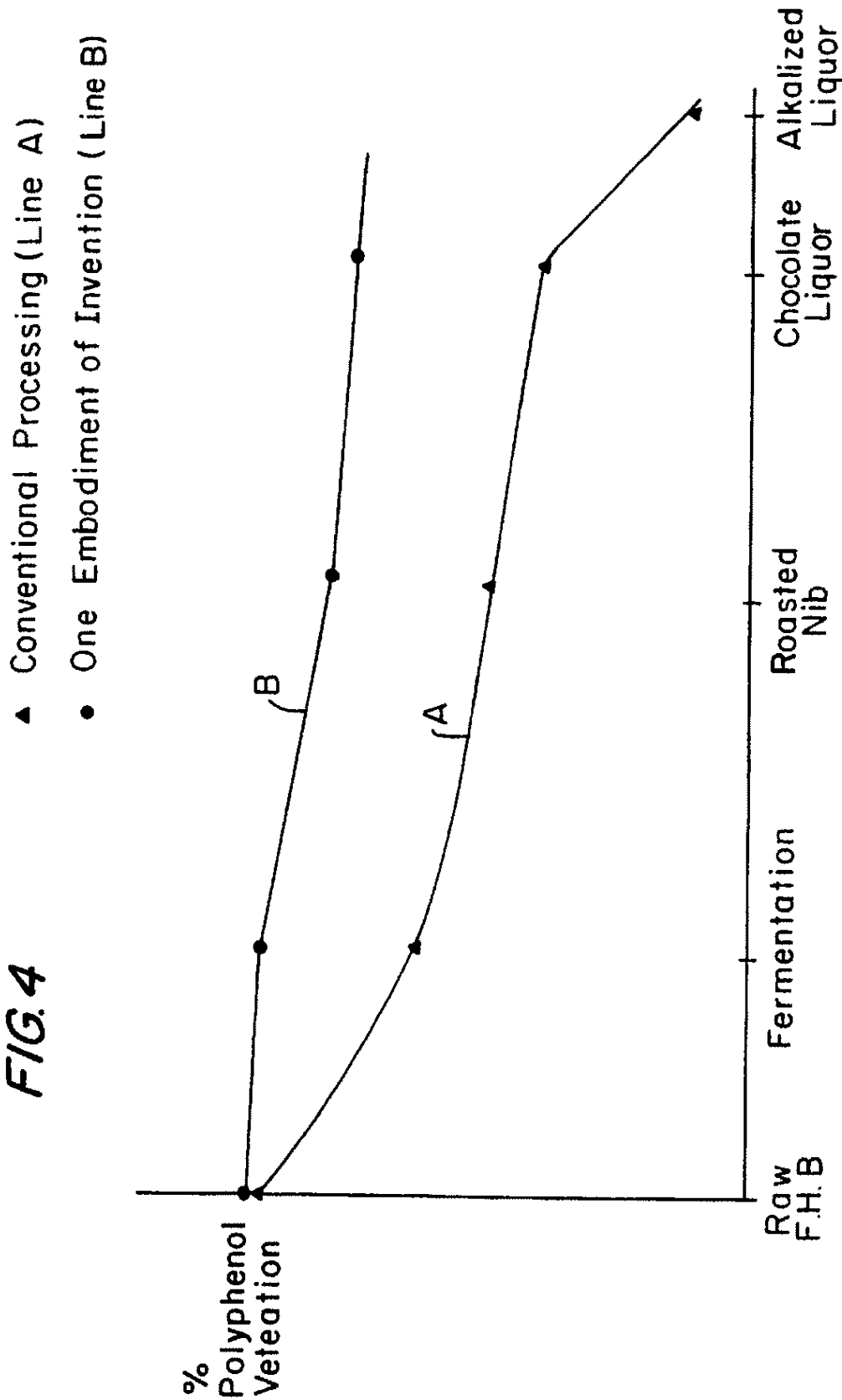
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*FIG. 3*

FIG. 4



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## Liquor Hold Study at Elevated Temperatures

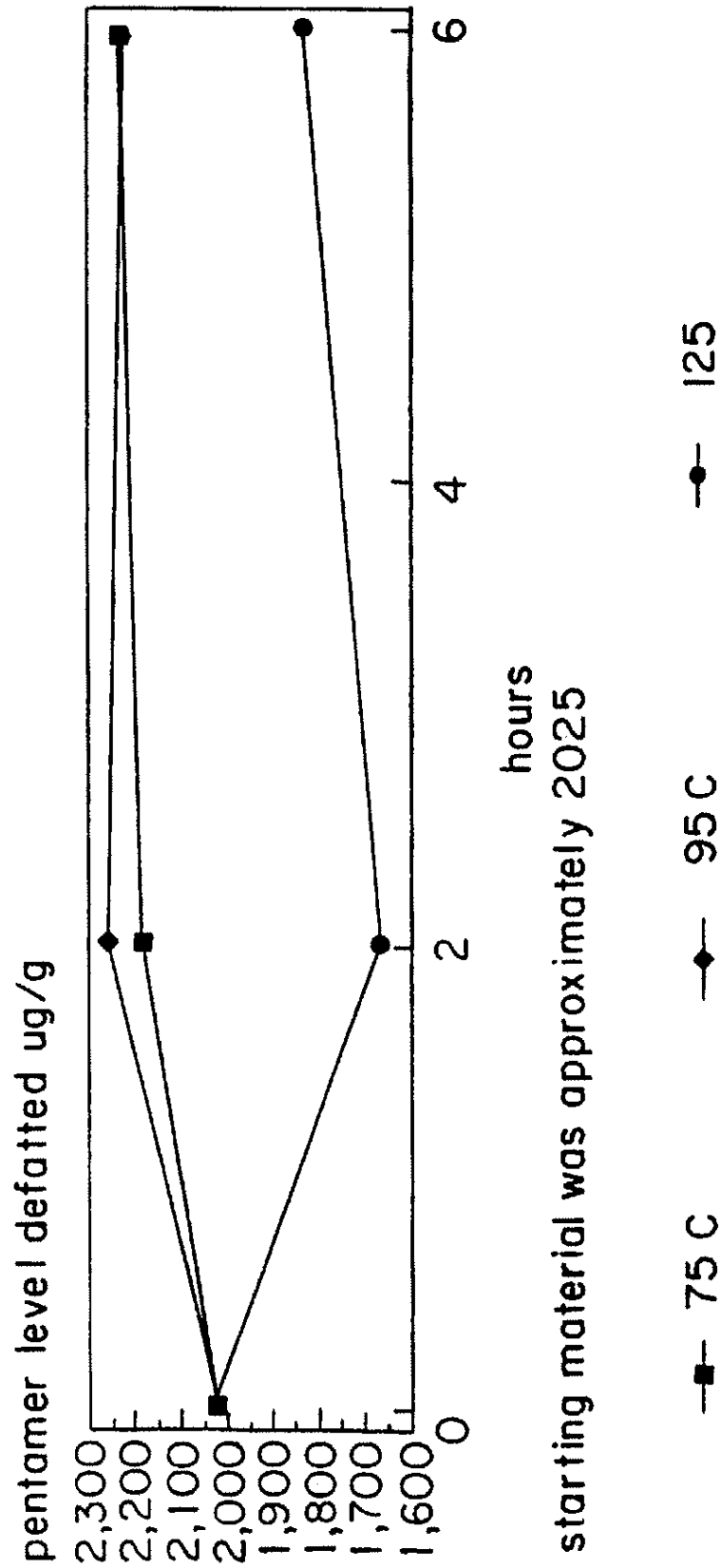


FIG.5



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# **COCOA COMPONENTS, EDIBLE PRODUCTS HAVING ENRICHED POLYPHENOL CONTENT, METHODS OF MAKING SAME AND MEDICAL USES**

This application is a continuation of U.S. application Ser. No. 09/041,326, filed Mar. 12, 1998, now U.S. Pat. No. 6,194,020, which was the National Stage of International application Ser. No. PCT/US97/15893, filed Sep. 8, 1997 and a continuation of U.S. Ser. No. 08/709,406, filed Sep. 6, 1996, now U.S. Pat. No. 6,015,913.

## **CROSS-REFERENCE TO RELATED APPLICATIONS**

Reference is made to copending U.S. applications Ser. No. 08/317,226, filed Oct. 3, 1994 (allowed, now U.S. Pat. No. 5,554,645), Ser. No. 08/631,661, filed Apr. 2, 1996, Ser. No. 08/709,406, filed Sep. 6, 1996, now U.S. Pat. No. 6,015,913 and Ser. No. 08/831,245, filed Apr. 2, 1997, incorporated herein by reference.

## **BACKGROUND OF THE INVENTION**

### **1. Field of the Invention**

The invention relates to cocoa components having enhanced levels of cocoa polyphenols, processes for producing the same, methods of using the same and compositions containing the same. More specifically, the invention provides a method of producing cocoa components having an enhanced content of cocoa polyphenols, in particular procyanidins. The cocoa components include partially and fully defatted cocoa solids, cocoa nibs and fractions derived therefrom, cocoa polyphenol extracts, cocoa butter, chocolate liquors, and mixtures thereof.

The invention also relates to versatile novel processes for extracting fat from cocoa beans and/or processing cocoa beans to yield a cocoa component having a conserved level of polyphenols, in particular procyanidins. The invention provides a significantly less complex process with respect to total cost of process equipment, maintenance, energy and labor, with the concomitant benefit of obtaining components having conserved concentrations of polyphenols relative to the starting materials.

### **2. Description of the Related Art**

Documents are cited in this disclosure with a full citation for each. These documents relate to the state-of-the-art to which this invention pertains, and each document cited herein is hereby incorporated by reference.

Confections and other edible compositions containing cocoa components have a very distinct taste and mouthfeel that have been enjoyed by individuals for many years. The unique flavor and mouthfeel of chocolate, for example, is a result of the combinations of its numerous components as well as its process of manufacture. It is well known that the mouthfeel and aroma/flavor of a chocolate are factors which greatly influence the desirability of the final chocolate product. Accordingly, the primary focus of conventional processes using cocoa components is the development of the distinctive chocolate mouthfeel and flavor/aroma. Throughout the entire chocolate manufacturing process, from the selection of the cocoa beans as a commodity at the country of origin to the tempering and solidification of the final chocolate, the development of the appropriate mouthfeel and/or aroma/flavor of the final product dictates the selections made and the process parameters used.

Chocolate contains solid particles dispersed throughout a fat matrix. Factors that influence the mouthfeel of a choco-

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late include the particle size distribution of the solids, the properties of the fat matrix material and how the chocolate is made.

Cocoa butter is typically the predominant fat in the chocolates. Cocoa butter is solid at room temperature (21°–24° C.) and thus most chocolates are firm and solid at room temperature providing good "snap" at initial bite. Above room temperature, the fat phase melts progressively until it is completely melted at about 36° C. This rapid melting in the mouth, at body temperature, provides the smooth, creamy mouthfeel which results in a strong flavor impact.

The flavor/aroma characteristics of the cocoa product are dependent on the combination of numerous solid and fat components as well as the process of manufacture. The flavor/aroma characteristics are dependent on (1) initial cocoa bean selection (i.e., level of fermentation, genotype, origin, etc.), (2) method of processing the beans (i.e., cleaning, roasting, shell removal, etc.) (3) processing of the cocoa components (i.e., milling) and (4) final processing to form the final product (i.e., selection of cocoa component and other ingredients, conching, etc.).

The several roles of selecting beans, fermenting them, cleaning them and processing them to obtain good flavor and other desirable characteristics is well known and is described below.

#### **1. The Cocoa Bean**

Cocoa beans are derived from cocoa trees which are found in warm, moist climates in areas about 20 degrees latitude north and south of the Equator. In general, the seeds of the *Theobroma cacao* (of the order Sterculiaceae) are known chiefly in two varieties: Criollo and Forastero, with Forastero divided into several varieties. A third group, called Trinitario, is essentially a cross between Criollo and Forastero and is not found in the wild. Freshly harvested raw Criollo beans are pale brown in color while Forastero beans are a purple hue.

The cocoa bean is comprised of an inner nib portion covered by an outer shell. After conventional drying, the shell of the bean comprises about 12 to 15% of the weight of the bean, while the nib and residual moisture amounts to approximately 85 to 88%. Typical analytical data ranges for chemical components of cocoa nib are: fat content of 48 to 57%; theobromine content of 0.8 to 1.3%; caffeine content of 0.1 to 0.7%; total nitrogen content of 2.2 to 2.5%; ash content of 2.6 to 4.2%; and water content of 2.3 to 3.2% (see *Pearson's Composition and Analysis of Foods*, 9th Ed., 1991).

#### **2. Fermentation of the Bean**

Fermentation, an early step in the processing of cocoa beans, is important to the development of suitable flavors and/or flavor precursors. It was previously believed that fermentation and drying of the cocoa beans were "of vital importance as no subsequent processing of the bean will correct that practice at this stage" (*Chocolate, Cocoa and Confectionery: Science and Technology*, 3rd Ed., by Bernard W. Minifie, p. 13 (1989)). During the fermentation and drying processes, the unfermented wet beans taken from the pod lose about 65 percent of their weight, assuming the final optimum moisture content of 6 percent is attained (Minifie, p. 14). The level of fermentation in the dried cocoa bean is typically determined by the "cut test" (defined further below).

It is well known in the art that flavor in the final cocoa or chocolate is closely related to fermentation. For example, if the beans are cleaned and separated from the pulp and dried without any fermentation, the nib will not be the brown or

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purple-brown color of fermented dry cocoa beans but instead a slaty grey color (*Industrial Chocolate Manufacture and Use*, 2nd Ed., by S. T. Beckett, p. 13). Chocolate made from slaty, unfermented beans typically tastes very bitter and astringent without any apparent chocolate flavor (Beckett, p. 13).

Accordingly, fully fermented cocoa beans are more desirable than underfermented cocoa beans from a flavor/aroma standpoint and typically sell at a higher price. The fermented cocoa beans are usually used to produce chocolate liquors.

Underfermented beans are conventionally processed for their cocoa butter. The quality of the cocoa butter is not affected by underfermentation. The quality of the cocoa solids, however, is affected since they do not contain sufficient color, flavor/aroma and are therefore either discarded or sold for low-value uses. Although chocolate liquors and/or partially defatted cocoa solids are sometimes produced from a nonhomogeneous mass of beans containing a portion of underfermented beans, the resultant liquor or solids require subsequent treatment or processing. Since unfermented beans are not conventionally processed commercially, they are not typically available.

### 3. Bean Cleaning

Once the cocoa beans are selected, they are cleaned to remove extraneous matter and then processed. The initial step consists of cleaning the beans to remove extraneous non-cocoa materials. Conventional bean cleaning separates beans from extraneous non-cocoa materials by either size or density using a cleaning machine which is a gravity, vibratory or aspiration table (see Minifie, p. 35; *Chocolate Production and Use*, 3rd Ed., by L. Russell Cook, pp. 144–146; and Beckett, p. 55).

Current cocoa bean cleaning technology is typically limited in separation ability to a minimum density difference of 10–15%. This reduces the efficiency of achieving an accurate separation of bean and extraneous non-cocoa materials and therefore reduces the clean bean yield of the process. Additionally, conventional cleaning machines become easily clogged and require frequent cleaning. This also reduces the cleaning efficiency and the clean bean yield of the overall process.

Moreover, conventional cleaning machines have a tendency to fracture the beans during cleaning which reduces the percentage of whole beans available after cleaning. These broken bean pieces can later give rise to problems during roasting and winnowing. For instance, small bean pieces will burn readily at the elevated temperatures used during roasting and may result in burnt and ashy flavored liquors which are unacceptable from a flavor standpoint. Small bean pieces may also decrease the efficiency of the winnowing process because they can be lost during the aspiration of the shells and result in overall yield efficiency losses.

### 4. Bean Roasting

In most conventional processes, roasting of the whole bean or nib is an essential step in the manufacture of chocolate liquor or partially defatted cocoa solids. Whole bean roasting was previously believed to be critical for developing the natural flavor and aroma of the cocoa beans and reducing the moisture content of the bean to below about 2% by weight. Whole bean roasting also loosens the shell so that it can be readily removed during the winnowing process. The degree of cocoa roast is a time/temperature dependent relationship, where the time can vary from 5 to 120 minutes and the temperature of the whole bean can typically vary from 120° C. to 150° C. In the pre-roasting of whole beans, an initial heating step can be performed at just below

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100° C., followed by roasting of the nibs at elevated temperatures up to about 130° C. (see Minifie, especially pp. 37 and 45–46; Cook, pp. 146–152; Beckett, pp. 55–64; and U.S. Pat. No. 5,252,349 to Carter, Jr.).

### 5. Winnowing-Shell Removal

The winnowing operation serves to separate the beans into the desired inner portion of the bean (nib) and the outer portion of the bean (shell). The principle of separation by a winnowing process depends on the difference in the apparent density of the nib and of the shell. Standard winnowing machines make use of the combined action of sieving and air aspiration. The shell is loosened during the conventional roasting and/or other heating steps. After loosening, the beans are typically broken between rollers to shatter the cocoa beans along natural fracture lines of the cocoa nib to facilitate shell removal during winnowing (see U.S. Pat. No. 2,417,078 to Jones; U.S. Pat. No. 5,252,349 to Carter, Jr.; Minifie, pp. 47–51; Cook, pp. 152–153; and Beckett, pp. 67–68).

Some cocoa bean processing techniques include a heat pre-treatment step to aid in the separation of the shell from the nib. This involves giving the beans a thermal shock by hot air, steam or infra-red heat (see U.S. Pat. No. 4,322,444 to Zuilichem et al.; British Patent No. 1,379,116 to Newton; Minifie, pp. 44–43; Cook, p. 155; and Beckett, pp. 60–62).

Infra-red heat pre-treatment uses infra-red heating to rapidly heat and expand the beans. This loosens the shells. The method consists of exposing the beans to infra-red radiation for a period of between one half and two minutes, during which time the beans are typically heated to a temperature of about 100 to 110° C. The infra-red radiation used has a wavelength between 2 and 6 microns which corresponds to a frequency in the range of 0.7 to 1.2×10<sup>8</sup> megacycles per second.

### 6. Formation of Chocolate Liquor and other Cocoa Components

The next step in conventional cocoa processing, after winnowing, involves nib grinding. Nib grinding is typically performed in two stages, an initial grinding stage to convert the solid nibs into a fluid paste and a final grinding stage to achieve the desired particle size. Both of these stages are equipment, maintenance, and energy intensive.

The cleaned roasted cocoa nibs typically vary in cocoa butter content from 50–58% by weight. During the grinding, the nib is ground, for instance by milling, into a fluid, dark brown “liquor”. The fluidity is due to the breakdown of the cell walls and the release of the cocoa butter during the processing. Ground particles of partially defatted cocoa solids are suspended in the cocoa butter. This liquor is sometimes commercially sold as a product useful in the confectionery and baking industries where machinery for processing the cocoa beans is not available.

Other conventional cocoa processing includes separating cocoa butter from liquor. This is accomplished by using a batch hydraulic pot press (“hydraulic press”) to separate the cocoa butter from the cocoa solids. The resultant cocoa butter is subsequently filtered to yield a clear, solid-free cocoa butter. Butter can also be produced by a continuous screw press to extract the butter from whole bean with shell or less frequently, from nibs (see U.S. Pat. No. 5,252,349 to Carter, Jr.; and Minifie, especially pp. 71–72).

The resulting cocoa cake from either hydraulic presses or screw presses may be milled into cocoa powder. Cocoa cake typically contains either 10–12% cocoa fat or 20–22% cocoa fat (see Minifie, pp. 72–76; Cook, pp. 169–172; and Beckett, pp. 78–82). Cocoa powder from cocoa cake obtained by hydraulic pressing is usually produced by milling the cocoa

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cake. If natural cocoa powder is desired, cocoa cake is fed directly to the cocoa cake mill. If alkalized cocoa powder is desired, the cake from an alkalizing process is fed to the mill. Hydraulic pressing produces a cocoa cake which is an agglomerate of previously milled cocoa particles. Cocoa cake mills for cocoa cake from hydraulic pressing are therefore designed to reduce the size of these agglomerates.

The natural cocoa cake or natural cocoa powder can be further processed by alkalizing to modify the color and flavor qualities of the cake (see U.S. Pat. No. 3,997,680 to Chalin; U.S. Pat. No. 5,009,917 to Wiant, et al.; Minifie, pp. 61-67; Cook, pp. 162-165; and Beckett, pp. 71-72). The alkalizing process can be used at any of several different stages of processing and includes the treatment of either the beans, liquor, nib, cake or powder with solutions or suspensions of alkali, usually, but not limited to, sodium or potassium carbonate. After alkalizing, the cocoa solids are dried and cooled. The dried cocoa solids are subsequently milled to produce alkalized cocoa powder, and thereafter cooled and packaged.

#### 7. Polyphenols in Cocoa Beans and Their Utility

Cocoa beans contain polyphenols. These polyphenols have recently been extracted and screened for biological activity. It has been discovered that cocoa polyphenol extracts, particularly procyanidins, have significant biological utility. The extracts or compounds further separated therefrom have generally been prepared, on a laboratory scale, by reducing cocoa beans to a powder, defatting the powder, and extracting and purifying the active compound(s) from the defatted powder. The powder is generally prepared by freeze-drying the cocoa beans and pulp, depulping and deshelling the freeze-dried beans and grinding the deshelled beans or nibs. The extraction of active compound(s) has been accomplished by solvent extraction techniques, and the extracts have been purified by gel permeation chromatography, preparative High Performance Liquid Chromatography (HPLC) techniques, or by a combination of such methods (see U.S. Pat. No. 5,554,645 to Romanczyk et al.).

It has now been determined that the recovery of polyphenols appears to be inversely proportional to the degree of fermentation of the cocoa beans. Accordingly, the use of fermented beans as a feedstock material, which is important for good chocolate flavor, reduces the amount of polyphenols available in the cocoa component(s) derived from the beans.

It has also been determined that higher processing temperatures and/or longer processing times, e.g. in the roasting step, reduces the amount of polyphenols available in the cocoa components derived from the feedstock beans. Cocoa components have not, heretofore, been produced having substantial quantities of polyphenols. These problems in the art have not heretofore been recognized.

#### OBJECTS OF THE INVENTION

It is an object of the invention to overcome the above-mentioned difficulties and/or deficiencies in the prior art.

More specifically, it is an object of the invention to provide methods of selecting and/or processing cocoa beans for producing cocoa components having enhanced levels of cocoa polyphenols.

It is a further object of the invention to provide a method of processing cocoa beans, wherein a significant amount of cocoa polyphenols present in the pre-processed bean is conserved in the processed bean.

It is yet another object of the invention to provide cocoa components, including cocoa nibs or portions thereof,

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chocolate liquor and partially or fully defatted cocoa solids, each having enhanced levels of cocoa polyphenols, and products containing the cocoa components.

It is an additional object of the invention to provide a method of manufacturing chocolates, chocolate-flavored confections, chocolate-flavored compositions, edible compositions, supplements, and combinations thereof having enhanced levels of cocoa polyphenols or derivatives thereof.

It is a further object of the invention to provide a method of improving the health of a mammal using the products of the invention.

It is a still further object of the invention to provide a method of improving the flavor/aroma characteristics of cocoa components, particularly chocolate liquor, containing enhanced levels of cocoa polyphenols.

It is a still further object of the invention to provide a method of producing cocoa butter and cocoa solids having a high yield of cocoa butter per amount of cocoa beans processed.

It is another object of the invention to provide a method of winnowing beans to remove the shell portion from the inner portion using an air fluidized bed density separation system.

It is another object of the invention to provide a method of producing high quality cocoa butter without requiring a bean roasting step or a liquor milling step.

These and other objects and advantages of the invention will become further apparent from the teachings hereinafter provided by the detailed description, test data, and examples.

#### SUMMARY OF THE INVENTION

The invention relates to novel versatile methods of processing cocoa beans to form cocoa components having improved properties or characteristics, products made from those methods and methods of using the same. More specifically, the invention relates to methods of producing cocoa components having enhanced levels of cocoa polyphenols. Parameters of the several cocoa processing steps, including the selection of the cocoa bean feedstock, are controlled and/or manipulated to result in a valuable cocoa component while conserving a significant amount of the cocoa polyphenol content present in the cocoa bean. Thus, the invention relates to methods of obtaining cocoa components having conserved levels of cocoa polyphenols relative to the starting materials, and to the products of those processes produced thereby. The invention avoids the significant and detrimental losses of cocoa polyphenols that occur during conventional processing.

The invention also relates to novel cocoa components having enhanced levels of cocoa polyphenols produced by the methods of the invention. More specifically, the invention relates to novel cocoa components including cocoa nibs or portions thereof, chocolate liquors, partially or fully defatted cocoa solids, cocoa polyphenol extract, and combinations thereof having higher levels of cocoa polyphenols in comparison with conventionally produced cocoa components.

The invention also relates to novel compositions containing the novel cocoa components including edible products, chocolates, chocolate-flavored confections, chocolate-flavored compositions, ingestible products, digestible products, chewable compositions and combinations thereof. The invention is thus in novel products having enhanced



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levels of cocoa polyphenols and novel products containing a cocoa polyphenol additive or a derivative thereof. The additive may be an extract from cocoa beans or a cocoa component thereof, or may be synthetic.

The invention further relates to the treatment of cocoa components, particularly chocolate liquors, to provide a cocoa component having high levels of cocoa polyphenols with acceptable aroma/flavor characteristics. The treatment includes the removal of undesirable and/or off flavors that may be present in a cocoa component, the manipulation of the aroma/flavor profile using additives or the blending of cocoa components having varying levels of cocoa polyphenols and varying degrees of aroma/flavor.

The invention also relates to methods for the production of cocoa polyphenol extract from cocoa beans or components thereof and to the use of the extract as an additive to edible compositions.

The invention also relates to novel methods of improving the health of a mammal, particularly humans, using the products containing cocoa polyphenols, particularly products containing elevated levels of cocoa polyphenols. These methods include the use of the cocoa polyphenols to provide one or more of the following activities: reducing periodontal disease, antigingivitis, antiperiodontitis, reducing atherosclerosis, LDL oxidation inhibitor, reducing hypertension, antineoplastic, antioxidant, DNA topoisomerase II enzyme inhibitor, cyclo-oxygenase modulator, lipoxygenase modulator, nitric oxide (NO) or NO-synthase modulator, non-steroidal anti-inflammatory, apoptosis modulator, platelet aggregation modulator, blood or in vivo glucose modulator, antimicrobial and inhibitor of oxidative DNA damage activity.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIGS. 1(a)-(d) illustrate the change in the surface of the cut bean half during the fermentation of the cocoa bean: FIG. 1(a) depicts the cut bean half of an unfermented cocoa bean; FIGS. 1(b)-(d) depict the cocoa bean as it is fermented, with FIG. 1(d) illustrating the fully fermented cocoa bean;

FIG. 2 is a graphical representation of cocoa polyphenols level/fermentation relationship for five cocoa bean samples, wherein the vertical axis represents the level of cocoa polyphenol pentamer (ug/g) from chocolate liquors derived from these cocoa beans defatted and the horizontal axis is the degree of fermentation using the fermentation factor (as defined below);

FIG. 3 shows an overview of the method of the present invention, and the various products which can be produced by the process (process options dependent upon economics of products, and/or by-products);

FIG. 4 is a graphical representation illustrating the levels of total cocoa polyphenols present in the cocoa bean or portion thereof during conventional chocolate liquor processing (line A) and during processing according to one embodiment of the invention (line B);

FIG. 5 is a graphical representation of cocoa polyphenols level/heating temperature/heating time relationship for chocolate liquor samples heat treated at three different temperatures, wherein the vertical axis represents the level of cocoa polyphenol pentamer (ug/g) from defatted chocolate liquors and the horizontal axis is the time of heat treatment.

#### DESCRIPTION OF THE PREFERRED EMBODIMENTS Definitions

1. The term "chocolate" refers to a solid or semi-plastic food and is intended to refer to all chocolate or chocolate-

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like compositions containing a dispersion of solids within a fat phase. The term is intended to include compositions conforming to the U.S. Standards Of Identity (SOI), CODEX Alimentarius and/or other international standards and compositions not conforming to the U.S. Standards Of Identity or other international standards. The term includes sweet chocolate, bittersweet or semisweet chocolate, milk chocolate, buttermilk chocolate, skim milk chocolate, mixed dairy product chocolate, sweet cocoa and vegetable fat coating, sweet chocolate and vegetable fat coating, milk chocolate and vegetable fat coating, vegetable fat based coating, pastels including white chocolate or coating made with cocoa butter or vegetable fat or a combination of these, nutritionally modified chocolate-like compositions (chocolates or coatings made with reduced calorie ingredients) and low fat chocolates, unless specifically identified otherwise.

In the United States, chocolate is subject to a standard of identity established by the U.S. Food and Drug Administration (FDA) under the Federal Food, Drug and Cosmetic Act. Definitions and standards for the various types of chocolate are well established in the U.S. Nonstandardized chocolates are those chocolates which have compositions that fall outside the specified ranges of the standardized chocolates.

The fat phase of the chocolate of the invention can include cocoa butter, milkfat, anhydrous milkfat, butteroil, and other vegetable fat and other modifications of these fats (CBR, CBE and CBS, referring to cocoa butter replacers, equivalents and substitutes) and synthetic fats or mixtures of cocoa butter with these fats. See Minifie, pp. 100-109.

The chocolate may contain a sugar syrup/solids, invert sugar, hydrolyzed lactose, maple sugar, brown sugar, molasses, honey, sugar substitute and the like. The term "sugar substitute" includes bulking agents, sugar alcohols (polyols such as glycerol), or high potency sweeteners or combinations thereof. Nutritive carbohydrate sweeteners with varying degrees of sweetness intensity may be any of those typically used in the art and include, but are not limited to, sucrose, e.g. from cane or beet, dextrose, fructose, lactose, maltose, glucose syrup solids, corn syrup solids, invert sugar, hydrolyzed lactose, honey, maple sugar, brown sugar, molasses and the like. Sugar substitutes may partially replace the nutritive carbohydrate sweetener. High potency sweeteners include aspartame, cyclamates, saccharin, acesulfame-K, neohesperidin dihydrochalcone, sucralose, alitame, stevia sweeteners, glycyrrhizin, thaumatin and the like and mixtures thereof. The preferred high potency sweeteners are aspartame, cyclamates, saccharin, and acesulfame-K. Examples of sugar alcohols may be any of those typically used in the art and include sorbitol, mannitol, xylitol, maltitol, isomalt, lactitol and the like.

The chocolates may also contain bulking agents. The term "bulking agents" as defined herein may be any of those typically used in the art and include polydextrose, cellulose and its derivatives, maltodextrin, gum arabic, and the like.

The chocolate products may contain emulsifiers. Examples of safe and suitable emulsifiers may be any of those typically used in the art and include lecithin derived from vegetable sources such as soybean, safflower, corn, etc., fractionated lecithins enriched in either phosphatidyl choline or phosphatidyl ethanolamine, or both, mono- and diglycerides, diacetyl tartaric acid esters of mono- and diglycerides (also referred to as DATEM), monosodium phosphate derivatives of mono- and diglycerides of edible fats or oils, sorbitan monostearate, hydroxylated lecithin, lactylated fatty acid esters of glycerol and propylene glycol,

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polyglycerol esters of fatty acids, propylene glycol mono- and di-esters of fats and fatty acids, or emulsifiers that may become approved for the US FDA-defined soft candy category. In addition, other emulsifiers that can be used include polyglycerol polyricinoleate (PGPR), ammonium salts of phosphatidic acid, (e.g. YN) sucrose esters, oat extract, etc., any emulsifier found to be suitable in chocolate or similar fat/solid system or any blend.

2. The term "chocolate-flavored confection" refers to food products, excluding "chocolate", having a chocolate flavor/aroma and comprising a cocoa fraction. These products are stable at ambient temperatures for extended periods of time (e.g., greater than 1 week) and are characterized as microbiologically shelf-stable at 18–30° C. under normal atmospheric conditions. Examples include chocolate-flavored hard candies, chewables, chewing gums, etc.

3. The term "chocolate-flavored compositions" refers to chocolate-flavored compositions, excluding "chocolate", containing a cocoa fraction and having a chocolate flavor/aroma. Examples include chocolate-flavored cake mixes, ice creams, syrups, baking goods, etc.

4. The term "fats", as used herein, refer to triglycerides, diglycerides and monoglycerides that can normally be used in chocolates and chocolate-like products. Fats include the naturally occurring fats and oils such as cocoa butter, pressed cocoa butter, expeller cocoa butter, solvent extracted cocoa butter, refined cocoa butter, milkfat, anhydrous milkfat, fractionated milkfat, milkfat replacers, butterfat, fractionated butterfat, cocoa butter equivalents (CBE), cocoa butter substitutes (CBS), cocoa butter replacers (CBR), reduced calorie fats and/or synthetically modified fats such as Caprenin®. An example of a reduced calorie fat is Caprocarylobehein (commonly known as Caprenin®) as described in U.S. Pat. No. 4,888,196 to Ehrman, et al., which is incorporated herein by reference.

5. The term "food product" includes any food product, for example, those set forth in 21 CFR § 101.12. The term includes chocolate-flavored compositions (e.g., cakes, nougats, puddings, etc.), as well as compositions not having a chocolate-flavor (e.g., caramels, etc.)

6. The term "fermentation factor" is a numerical quantification of the level of fermentation of a batch of cocoa beans. Fermentation factors range from 100 (under/unfermented) to 400 (fully fermented).

In order to assess the degree of fermentation, cocoa beans are typically subjected to a standard cut test for assessing quality as defined in industry grade standards. The bean halves are laid out on a board for visual inspection of color as well as defects which can arise during bean fermentation, drying and/or storage.

Beans can be divided into four fermentation categories according to their color and appearance: (a) fully fermented, e.g., predominantly a brown hue; (b) partially fermented, e.g., purple/brown; (c) purple (underfermented); and (d) slaty (very underfermented and/or unfermented beans).

Purple/brown beans include all beans showing any blue, purple or violet color on the exposed surface, whether suffused or as a patch. Purple beans should include all beans showing a completely blue, purple or violet color over the whole exposed surface. This should also include, irrespective of color, any beans which are slaty, but not predominantly so (wherein predominantly, in this context, means more than half).

The "fermentation factor" is determined using a grading system for characterizing the fermentation of the cocoa beans. Slaty, being under/unfermented, is designated as 1,

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purple as 2, purple/brown as 3 and brown as 4. The percentage of beans falling within each category is multiplied by the weighted number. Thus, the "fermentation factor" for a sample of 100% brown beans would be 100x4 or 400, whereas the fermentation factor for a sample of 100% purple beans would be 100x2 or 200. A sample of 50% slaty beans and 50% purple beans would have a fermentation factor of 150 [(50x1)+(50x2)].

Cut tests applicable to cocoa beans derived from the Trinitario and Forastero types may or may not be applicable to cocoa beans derived from the Criollo type, for example, where bean color variation ranging from fully purple to light tan can be encountered. Accordingly, the cut test based on color would not be applicable to specific cocoa genotypes lacking the anthocyanin pigments responsible for the purple color, such as the Catango (or Catongo) type whose beans are light tan in color. Other exceptions include "cocoa beans" derived from other Theobroma species, the Herrania species and their inter- and intra-specific crosses. The beans from these species are "tan" in color. For these types of beans the level of fermentation may be determined using a modified standard cut test. Using the modified test, the surface of the bean (halved) is inspected for the degree of lines, fissures or cracks which form during fermentation, rather than the change of color.

FIGS. 1(a)–(d) illustrate the change in the surface of the cut bean half during the fermentation of the cocoa bean. As can be seen from FIGS. 1(a)–(d), the number of lines/fissures and the extent to which they extend across the entire surface of the cut bean half increases as the bean is fermented. FIG. 1(a) depicts the cut bean half of an unfermented cocoa bean where the surface is relatively smooth. FIGS. 1(b)–(d) depict the cocoa beans as it is fermented, with FIG. 1(d) illustrating the fully fermented cocoa bean. As the cocoa bean is fermented, the surface develops small branch-like lines or fissures. This modified test can also be used to approximate the fermentation factor wherein a cocoa bean corresponding to FIG. 1(a) is designated as 100, FIG. 1(b) as 200, FIG. 1(c) as 300 and FIG. 1(d) as 400.

While the definitions of the aforementioned categories are a general guide, the assessment according to these categories is well within the skill of the ordinary skilled artisan well versed in chocolate and cocoa processing (see Wood et al., *Cocoa*, 4th Ed. (1985), incorporated herein by reference, especially pages 511 to 513).

7. The numerical terms or qualitative characteristics of the level of cocoa polyphenols in beans or in components refer to the amount detectable and measurable using the method of evaluating the levels set forth in Example 5.

8. The term "significant amount" means an amount which maintains the basic characteristics of the specified ingredients or composition or product.

9. The term "chocolate liquor" refers to the dark brown fluid "liquor" formed by grinding a cocoa nib. The fluidity is due to the breakdown of the cell walls and the release of the cocoa butter during the processing resulting in a suspension of ground particles of cocoa solids suspended in cocoa butter (See, *Chocolate, Cocoa and Confectionery: Science and Technology*, 3rd Ed., by Bernard W. Minifie).

10. The term "fair average quality cocoa beans" refers to cocoa beans that have been separated from the pulp material and dried and are relatively free of mold and infestation. Such beans are a commercial commodity and form the feedstock for the next step in the production processes, i.e. infra-red heating, roasting, pressing, etc. The term includes any such bean that has been genetically modified or produced.

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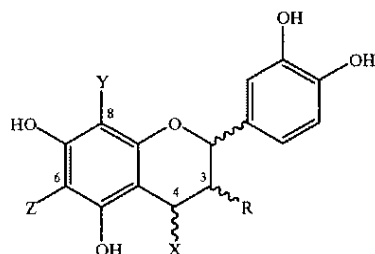
11. The term "raw freshly harvested cocoa beans" refers to seeds or beans freshly harvested from the cocoa pod and which have not been subjected to processing other than separation from the pulp. The term includes any such bean that has been genetically modified or produced.

12. The term "partially defatted cocoa solids" refers to the solid portion(s) derived from shell-free partially defatted cocoa nibs, including cocoa powders, cocoa cake, cocoa polyphenol extracts, alkalized powders or cakes, etc. (excluding chocolate liquor and cocoa butter).

13. The term "cocoa polyphenol" refers to the polyphenol compounds present in cocoa beans and derivatives thereof. The term cocoa polyphenol is intended to include polyphenols extracted from cocoa beans and derivatives thereof, as well as structurally similar synthetic materials.

The term polyphenols includes the proanthocyanidins extracted from cocoa beans and derivatives thereof, as well as structurally similar synthetic materials and includes the procyanidins extracted from cocoa beans and derivatives thereof as well as structurally similar synthetic materials.

More specifically, the term "cocoa polyphenol" includes monomers (notwithstanding the term polyphenol) of the formula  $A_n$  (where n is 1) or oligomers of the formula  $A_n$  (where n is an integer from 2 to 18, and higher), wherein A has the formula:



and R is 3-( $\alpha$ )-OH, 3-( $\beta$ )-OH, 3-( $\alpha$ )-O-sugar, or 3-( $\beta$ )-O-sugar;

bonding between adjacent monomers takes place at positions 4, 6 or 8;

a bond to a monomer in position 4 has alpha or beta stereochemistry;

X, Y and Z are selected from the group consisting of A, hydrogen, and a sugar, with the provisos that as to at least one terminal monomer, bonding of the adjacent monomer thereto is at position 4 and optionally Y=Z=hydrogen;

the sugar is optionally substituted with a phenolic moiety; and

salts, derivatives and oxidation products thereof.

Advantageously, the sugar is selected from the group consisting of glucose, galactose, xylose, rhamnose and arabinose. The sugar of any or all of R, X, Y, and Z can optionally be substituted at any position with a phenolic moiety via an ester bond. The phenolic moiety is selected from the group consisting of caffeine, cinnamic, coumaric, ferulic, gallic, hydroxybenzoic and sinapic acids. One or more of the cocoa polyphenol compounds may be used simultaneously, e.g., in "combinations" in formulation(s) comprising one or more of such compounds.

The term "oligomer", as used herein, refers to any compound of the formula presented above, wherein n is 2 through 18, and higher. When n is 2, the oligomer is termed a "dimer"; when n is 3, the oligomer is termed a "trimer";

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when n is 4, the oligomer is termed a "tetramer"; when n is 5, the oligomer is termed a "pentamer"; and similar recitations may be designated for oligomers having n up to and including 18 and higher, such that when n is 18, the oligomer is termed an "octadecamer".

The term "cocoa polyphenols" is further defined in U.S. applications Ser. No. 08/317,226, filed Oct. 3, 1994 (allowed, now U.S. Pat. No. 5,554,645), Ser. No. 08/631,661, filed Apr. 2, 1996, Ser. No. 08/709,406, filed Sep. 6, 1996, and Ser. No. 08/831,245, filed Apr. 2, 1997, incorporated herein by reference.

14. The term "treating" is intended to refer to methods of processing the cocoa beans including drying, heating (e.g., roasting, infra-red heating, etc.), chemical treatment (e.g., with anti-microbial agents), rehydrating, pressing, solvent extraction, microwave assisted extraction, etc.

15. The term "cocoa component" is intended to refer to a fraction derived from shell-free cocoa nib and includes chocolate liquor, partially or fully defatted cocoa solids (e.g., cake or powders), cocoa extracts, cocoa butter, cocoa nib or portions thereof, etc.

## DETAILED DESCRIPTION OF THE INVENTION

### A. Cocoa Bean selection

As set forth above, conventional processes utilize fermented cocoa beans to form cocoa components. Applicants have discovered that the level of cocoa polyphenols in the cocoa beans decreases dramatically during fermentation. FIG. 2 shows the pentamer content of liquors derived from cocoa beans of different origins with varying degrees of fermentation. The data represented in this graph were collected by visually color sorting the beans. Categories used in grading were slaty, purple, purple brown, and brown—the standard categories used by the industry to grade fermentation levels of beans during a cut test. Each sample (300 g) was roasted for 15 minutes at 150° C. in a convection oven. The roasted beans were then cracked and winnowed. A liquor was produced using a Melange milling apparatus with a one hour cycle time. To make the fermentation a continuous scale (x-axis) the different colors were given a weighted number.

These results demonstrated that underfermented beans have higher polyphenol levels than fermented beans. By processing underfermented beans it is possible to make liquors with higher polyphenol contents.

Accordingly, one aspect of the invention relates to methods of producing cocoa components containing enhanced levels of cocoa polyphenols from underfermented cocoa beans. The use of underfermented cocoa beans or a blend of underfermented cocoa beans with fermented cocoa beans provides a cocoa component having enhanced levels of cocoa polyphenols.

Therefore, one embodiment of the invention relates to the use of cocoa beans having a fermentation factor less than 375, preferably less than 325, advantageously less than 275, even more advantageously less than 225, desirably less than 175 and most desirably less than 150. In another preferred embodiment underfermented cocoa beans having a fermentation factor less than 125 and even about 100 are used.

### B. Methods of Producing Cocoa Components Having Enhanced Levels of Polyphenols

An outline of one embodiment of the invention is shown in FIG. 3. The method of the invention includes modifica-



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tions of certain steps of the process to produce three types of products. One process modification (Modification A) enables the production of cocoa solids containing conserved levels of polyphenols relative to the level of polyphenols in the cocoa bean feedstocks. Polyphenols are conserved in the product at higher levels than in conventional processes. Modification B enables the production of cocoa butter without necessarily the concomitant conservation of polyphenols. Modification C enables the production of cocoa solids and fat products with enhanced contents of polyphenols relative to conventional solid/fat separation processes.

In a broad embodiment of the invention a cocoa component having an enhanced content of cocoa polyphenol, is produced in a process comprising the steps of:

- (a) treating cocoa beans containing cocoa polyphenols while conserving a significant amount of the cocoa polyphenols content thereof to form treated cocoa beans; and
- (b) producing the cocoa component from the treated cocoa beans.

A significant amount of the cocoa polyphenols is conserved using the inventive methods.

The cocoa beans may be fair average quality cocoa beans, raw freshly harvested cocoa beans or combinations thereof. The cocoa beans may be unfermented, underfermented, fully fermented or mixtures thereof, with fermentation factors ranging from 100 to 400. Preferably, the cocoa beans are underfermented to enable the production of a cocoa component having the highest levels of cocoa polyphenols.

One embodiment of the invention relates to methods of processing cocoa beans which are fair average quality cocoa beans wherein the cocoa polyphenols content of the cocoa component produced is from 25 to 100% by weight of the cocoa polyphenols content of the fair average quality cocoa beans. Preferably, the cocoa polyphenols content of the cocoa component produced is greater than 35% by weight of the cocoa polyphenols content of the fair average quality cocoa beans, advantageously greater than 45% by weight, even more advantageously greater than 55% by weight, and most advantageously greater than 65% by weight. According to other preferred embodiments, more than 75% by weight is conserved, desirably more than 85% by weight, more desirably more than 95% by weight and most desirably more than 99% by weight.

Another embodiment of the invention relates to methods of processing raw freshly harvested cocoa beans wherein the cocoa polyphenols content of the cocoa component produced is from 5 to 100% by weight of the cocoa polyphenols content of the raw freshly harvested cocoa beans. Preferably, the cocoa polyphenols content of the cocoa component produced is greater than 10% by weight of the cocoa polyphenols content of the raw freshly harvested cocoa beans, advantageously greater than 15%, more advantageously greater than 20%, and still more advantageously greater than 25%. According to one preferred embodiment, greater than 30% is conserved, advantageously greater than 35%, more advantageously greater than 40% and most preferred greater than 45%. According to a still further preferred embodiment, greater than 50% is conserved, advantageously greater than 55%, even better greater than 60% and most preferred greater than 65%. According to an even further preferred embodiment, greater than 70% is conserved, advantageously greater than 75%, even better greater than 80% by weight and most preferred greater than 85%.

The processing steps include heat-treating (e.g., roasting, infra-red heating, etc.), drying, chemical treatments, etc.

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Preferably, the treatment steps develop chocolate flavor without significantly reducing the cocoa polyphenols content of the feedstock thereof to form heat-treated cocoa beans.

According to one embodiment of the invention the step of treating the cocoa beans comprises heat-treating the cocoa beans at an elevated temperature for a time sufficient to develop chocolate flavor while conserving a significant amount of the cocoa polyphenols content thereof to form heat-treated cocoa beans.

The heat-treating includes roasting, infra-red heating, drying at elevated temperatures and combinations thereof.

According to one embodiment of the invention, the heating of the cocoa bean is to an IBT (internal bean temperature) greater than 120° C. for at least 1 minute and the content of cocoa polyphenols in the heat-treated beans is at least 75% by weight (fullfat) of the cocoa polyphenols content in the pre-treated cocoa beans, advantageously greater than 80% by weight, more desirable greater than 85% by weight, even better greater than 90% by weight and most preferred greater than 95% by weight.

According to another embodiment of the invention, the heating of the cocoa bean is to an IBT (internal bean temperature) above 140° C. for at least 1 minute and the content of cocoa polyphenols in the heat-treated beans is at least 60% (fullfat) by weight of the cocoa polyphenols content in the pre-treated cocoa beans, advantageously greater than 65% by weight, more desirable greater than 70%, even better greater than 75% and most preferred greater than 80%.

According to yet another embodiment of the invention, the heating of the cocoa bean is to an IBT (internal bean temperature) above 160° C. for at least 1 minute and the content of cocoa polyphenols in the heat-treated beans is at least 40% by weight (fullfat) of the cocoa polyphenols content in the pre-treated cocoa beans, advantageously greater than 45%, more desirable greater than 50% by weight, even better greater than 55% by weight and most preferred greater than 60%.

According to a still further embodiment of the invention, the heating of the cocoa bean is to an IBT (internal bean temperature) above 120° C. for at least 1 minute and the content of cocoa polyphenol pentamer (fullfat) in the heat-treated beans is at least 60% by weight of the cocoa polyphenol pentamer content in the pre-treated cocoa beans, advantageously greater than 65%, more desirable greater than 70%, even better greater than 75% and most preferred greater than 80%.

According to another embodiment of the invention, the heating of the cocoa bean is to an IBT (internal bean temperature) above 140° C. for at least 1 minute and the content of cocoa polyphenol pentamer (fullfat) in the heat-treated beans is at least 25% by weight of the cocoa polyphenol pentamer content in the pre-treated cocoa beans, advantageously greater than 30%, more desirable greater than 35%, even better greater than 40% and most preferred greater than 50%.

According to another embodiment of the invention, the heating of the cocoa bean is to an IBT (internal bean temperature) above 160° C. for at least 1 minute and the content of cocoa polyphenol pentamer (fullfat) in the heat-treated beans is at least 15% by weight of the cocoa polyphenol pentamer content in the pre-treated cocoa beans, advantageously greater than 20%, more desirable greater than 25%, even better greater than 30% and most preferred greater than 35%.

Roasting comprises applying external heat to the cocoa bean or nib by a combination of conduction and convection.

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With conventional roasting conditions, moisture and volatile substances diffuse from the inner parts of the nib pieces.

According to one embodiment of the invention, roasting is preferably conducted at an internal bean temperature of from 95 to 160° C. for from 30 seconds to 5 hours, advantageously from 95 to 150° C. for from 1 minute to 3 hours, even better from 95 to 140° C. for from 1 minute to 1 hour and most preferred from 95 to 120° C. for from 1 minute to 1 hour.

Infra-red heating comprises applying infra-red heat so that the shells of the beans are rapidly heated. The shells dry, expand and loosen themselves from the nibs.

Preferably, the infra-red heating is conducted at an internal bean temperature of from 95 to 135° C. for from 1 to 5 minutes, advantageously from 95 to 125° C., even better from 95 to 115° C. and most preferred from about 95–110° C.

Preferably, the infra-red heating step is for a period of time less than 8 minutes, advantageously less than 7 minutes, even better less than 6 minutes and most preferred less than 5 minutes. According to a preferred embodiment, the period of time is less than 4 minutes, advantageously less than 3 minutes, even better less than 2 minutes and most preferably less than 1 minute.

According to one embodiment, the treating comprises drying the cocoa beans to form dried cocoa beans. The drying may be at ambient temperature or at an elevated temperature, preferably for a time and to an extent sufficient to develop chocolate flavor while conserving a significant amount of the cocoa polyphenols content thereof. The drying typically reduces the moisture of the cocoa bean to less than 7% by weight. Preferably, the drying decreases the moisture content of the cocoa bean to less than 4% by weight, advantageously to less than 3% by weight, even better to less than 2% by weight and most preferred to less than 1% by weight.

This embodiment of the invention may further comprise the step of producing chocolate liquor containing an enhanced content of cocoa polyphenols from the dried cocoa beans. The chocolate liquor may be produced by conventional grinding methods. Preferably, the chocolate liquor is cooled during grinding to reduce further losses of cocoa polyphenols.

According to another embodiment, the cocoa beans are raw freshly harvested cocoa beans containing a cocoa polyphenols content and the treating comprises:

- (i) at least partially fermenting the raw freshly harvested cocoa beans to form at least partially fermented cocoa beans; and
- (ii) heat-treating the at least partially fermented cocoa beans at an elevated temperature for a time sufficient to develop chocolate flavor while conserving a significant amount of the cocoa polyphenols content thereof to form heat-treated cocoa beans.

Preferably, the cocoa beans are raw freshly harvested cocoa beans having a fermentation factor less than about 125.

According to another embodiment, the treating comprises:

- (i) drying cocoa beans containing a cocoa polyphenols content to form dried cocoa beans; and
- (ii) infra-red heating the dried cocoa beans at an elevated temperature for a time sufficient to form infra-red heated cocoa beans while conserving a significant amount of the cocoa polyphenols content thereof.

According to yet another embodiment, the cocoa beans have shells and the treating comprises:

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- (i) infra-red heating the cocoa beans at an elevated temperature for a time sufficient to loosen the shells while conserving a significant amount of the cocoa polyphenols content thereof to form infra-red heated cocoa beans; and

- (ii) roasting the infra-red heated cocoa beans at an elevated temperature for a time sufficient to develop chocolate flavor while further conserving a significant amount of the cocoa polyphenols content thereof to form roasted cocoa beans.

According to a still further embodiment, the treating comprises:

- (i) infra-red heating the cocoa beans at an elevated temperature for a time sufficient to reduce their moisture to less than 5% by weight while conserving a significant amount of the cocoa polyphenols content thereof to form infra-red heated cocoa beans; and

- (ii) roasting the infra-red heated cocoa beans at an elevated temperature for a time sufficient to develop chocolate flavor while further conserving a significant amount of the cocoa polyphenols content thereof to form roasted cocoa beans.

According to another embodiment of the invention, the treating comprises:

- (i) drying cocoa beans containing a cocoa polyphenols content to form dried cocoa beans;

- (ii) infra-red heating the dried cocoa beans at an elevated temperature for a time sufficient to develop chocolate flavor while conserving a significant amount of the cocoa polyphenols content thereof to form infra-red heated cocoa beans; and

- (iii) roasting the infra-red heated cocoa beans at an elevated temperature for a time sufficient to further-develop chocolate flavor while further conserving a significant amount of the cocoa polyphenols content thereof to form roasted cocoa beans.

Surprisingly, it has been discovered that the polyphenols content of the cocoa bean may be maintained or conserved by controlling the treatment of the beans. Referring to FIG. 4, a graphical representation illustrates the levels of total cocoa polyphenols present in the cocoa bean or portion thereof during conventional chocolate liquor processing (line A) and processing according to one embodiment of the invention (line B). As can be seen by the graph, an initial loss in polyphenols content occurs during fermentation, additional loss occurs during roasting and further loss occurs during liquor, nib, cake or powder alkalizing (during the manufacture of chocolate).

According to the invention, the cocoa polyphenols content of the cocoa component produced is from 25 to 100% by weight of the cocoa polyphenols content in the fair average quality cocoa beans, advantageously from 35 to 100% by weight, more desirable from 45 to 100% by weight, even better from 55 to 100% by weight and more preferred from 65 to 100% by weight.

The invention permits the retention of higher levels of the cocoa polyphenols content not only with respect to the fair average quality cocoa beans, but also with respect to raw freshly harvested cocoa beans. Using the method of the invention, the cocoa polyphenols content of the cocoa component produced is from 5 to 100% by weight of the cocoa polyphenols content in the raw freshly harvested cocoa beans, advantageously from 10 to 75% by weight of the cocoa polyphenols content in the raw freshly harvested cocoa beans, preferably from 15 to 50% by weight, even better from 20 to 45% by weight and most preferred greater than 30% by weight.



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According to one embodiment, the cocoa polyphenols content of the infra-red heated cocoa beans is at least 55% by weight of the cocoa polyphenols content of the fair average quality cocoa beans, preferably at least 65%, advantageously at least 75%, even better at least 85% and most preferred at least 95%.

The cocoa polyphenol pentamer content of the infra-red heated cocoa beans may be at least 30% by weight of the cocoa polyphenol pentamer content of the fair average quality cocoa beans, preferably at least 35%, advantageously at least 40%, even better at least 45% and most preferred at least 50%.

When infra-red heating and roasting steps are used in combination, the cocoa polyphenols content of the roasted cocoa beans is preferably at least 75% by weight of the cocoa polyphenols content of the infra-red heated cocoa beans, advantageously at least 80%, even better at least 85% and most preferred at least 90%. Alternatively, the cocoa polyphenol pentamer content of the roasted cocoa beans is at least 40% by weight of the cocoa polyphenol pentamer content of the infra-red heated cocoa beans, advantageously at least 50%, even better at least 60% and most preferred at least 70%.

One preferred aspect of the invention relates to the production of chocolate liquors containing enhanced levels of cocoa polyphenols. Therefore, the cocoa components produced by the inventive methods preferably include chocolate liquor.

Accordingly, one embodiment of the invention relates to a method for the production of chocolate liquor having an enhanced content of cocoa polyphenols comprising the steps of:

- (a) treating cocoa beans, containing cocoa polyphenols, while conserving a significant amount of the cocoa polyphenols content thereof to form treated cocoa beans; and
- (b) producing chocolate liquor containing an enhanced content of cocoa polyphenols from the treated cocoa beans.

Preferably, the cocoa polyphenols content in the chocolate liquor is at least 65% by weight of the cocoa polyphenols content of the cocoa beans, advantageously at least 75%, even better at least 85% and most preferred greater than 90%.

Preferably, the cocoa polyphenol pentamer content in the chocolate liquor is at least 45% by weight of the cocoa polyphenol pentamer content of the cocoa beans, advantageously at least 55%, even better at least 60% and most preferred greater than 75%.

The invention also relates to the treatment of cocoa components, particularly chocolate liquors, to provide a cocoa component having high levels of cocoa polyphenols with acceptable aroma/flavor characteristics. The treatment includes the removal of undesirable or off flavors present in a cocoa component. The flavor may also be modified using additives or the blending of cocoa components having varying levels of cocoa polyphenols and varying degrees of aroma/flavor.

The chocolate liquor or cocoa component may be subsequently heat-treated to remove any undesirable or off flavors.

The subsequent heat-treating is preferably to a temperature between 65 and 140° C. for from 5 minutes to 24 hours, advantageously between about 75 and 130° C. for from 5 minutes to 2 hours, even better between about 85 and 120° C. for from 5 minutes to 1 hour and most preferred between about 95 and 110° C. for from 5 minutes to 30 minutes.

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Preferably, the subsequent heat-treating includes agitation to facilitate the removal of off-flavors. The heating may be under a vacuum to assist in the removal of off-flavors, preferably wherein the pressure is less than 26 inches (660 mm) mercury. The chocolate liquor or cocoa component may also be aerated during the heat-treatment. FIG. 5 illustrates the effect of different heat treatment temperatures (75° C., 95° C., 125° C.) on pentamer level vs. time of heating in a chocolate liquor. FIG. 5 shows that long treatments at temperatures greater than 100° C. should be avoided.

According to one embodiment, the liquor or cocoa component is subsequently directly heated with steam.

Preferably, the cocoa polyphenols content of the chocolate liquor is at least 55% by weight of the cocoa polyphenols content of the cocoa beans, advantageously at least 65%, even better at least 75% and most preferred at least 85%.

Preferably, the cocoa polyphenol pentamer content of the chocolate liquor is at least 45% by weight of the cocoa polyphenol pentamer content of the cocoa beans, advantageously at least 55%, even better at least 65% and most preferred at least 75%.

Another aspect of the invention relates to methods of making chocolate liquors without the use of an alkalization step and/or without the use of a conventional roasting step.

One embodiment of the invention relates to methods of making a non-alkalized chocolate liquor comprising the steps of:

- (a) heating cocoa beans using infra-red radiation; and
- (b) producing a chocolate liquor from the heated cocoa beans; wherein the chocolate liquor is not subsequently alkalized.

Another embodiment of the invention relates to a method of making a chocolate liquor comprising the steps of:

- (a) heating cocoa beans using infra-red radiation to loosen their shell; and
- (b) producing a chocolate liquor from the heated cocoa beans without a subsequent heating step.

According to this embodiment of the invention, the heating is achieved by the use of an infra-red heater. A suitable infra-red heater is manufactured by Micronizer company (U.K.) Ltd. The infra-red heating is performed at elevated temperatures as compared to conventional processing conditions to not only assist in removing the strongly adhering shells from the cocoa nibs, but also to lightly roast the raw beans. The level of thermal processing achieved with the infra-red heating eliminates the need for a conventional bean roaster. The infra-red heating puffs and loosens the shells from the beans to facilitate removal in the winnowing process. Preferably, the infra-red heating is performed at elevated temperatures to give a sufficient roast to the raw beans and thereby eliminate the need for an additional bean roaster. The elimination of the conventional bean roasting step greatly simplifies and reduces the cost of the method or process.

Preferably, the heating reduces the cocoa bean moisture content to less than 7% by weight, preferably less than 5% by weight, advantageously less than 4%, even better less than 3%, and most preferred less than 2%.

As set forth above, the cocoa polyphenols content of the cocoa beans decreases dramatically during fermentation. One aspect of the invention relates to the use of underfermented or unfermented cocoa beans in the production of the cocoa component. Preferably, the cocoa beans have a fermentation factor less than 375, advantageously less than 350, even better less than 325, and most preferred less than 300.

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According to yet another embodiment, highly underfermented cocoa beans are used. Preferably, the cocoa beans have a fermentation factor less than 275, advantageously less than 250, even better less than 225, and most preferred less than 200. Cocoa beans having a fermentation factor less than 150 or even unfermented beans (i.e., a fermentation factor of about 100) may also be used.

According to another aspect of the invention, the method comprises the step of at least partially fermenting raw freshly harvested cocoa beans containing a cocoa polyphenols content to form at least partially fermented cocoa beans and subsequently treating the at least partially fermented cocoa beans. Preferably, the at least partially fermented cocoa beans have a fermentation factor less than 375, even better less than 200 and most preferred less than 150.

Another aspect of the invention relates to methods for the commercial production of cocoa polyphenols, for use as an edible, ingestible or pharmaceutical component, from cocoa beans comprising the steps of:

- (a) processing cocoa beans to separate cocoa butter from cocoa solids; and
- (b) extracting cocoa polyphenols from the cocoa solids, wherein the processing comprises the steps of pressing, microwave assisted extraction (see U.S. Pat. No. 5,002,784 to Pare et al.), solvent extraction or combinations thereof.

Another embodiment of the invention relates to methods for the commercial production of cocoa polyphenols from cocoa beans comprising the sequential steps of:

- (a) extracting cocoa polyphenols from the cocoa beans; and
- (b) separating a cocoa component from cocoa shell.

According to one preferred embodiment, the cocoa beans are underfermented to enhance the amount of cocoa polyphenols. Preferably, the cocoa beans have a fermentation factor less than 375, even better less than 350 and most preferred less than 325.

#### C. Cocoa Components having Enhanced Levels of Cocoa Polyphenols

##### 1. Chocolate Liquors

Using the above-described methods, chocolate liquors having enhanced levels of cocoa polyphenols are obtained.

When characterizing an inventive product by relating the amount of cocoa polyphenols per gram ingredient in the inventive product, that ingredient does not necessarily contain the cocoa polyphenols, but rather it is the product that contains the cocoa polyphenols.

One embodiment relates to a chocolate liquor produced from fair average quality cocoa beans having a fermentation factor greater than 375, the chocolate liquor containing at least 5500  $\mu\text{g}$ , preferably at least 6000  $\mu\text{g}$ , advantageously at least 7000  $\mu\text{g}$ , even better at least 8000  $\mu\text{g}$  and most preferred at least 9000  $\mu\text{g}$  cocoa polyphenols per gram chocolate liquor. Preferably, the chocolate liquor contains at least 500  $\mu\text{g}$  cocoa polyphenols pentamer per gram chocolate liquor, advantageously at least 600  $\mu\text{g}$ , even better at least 700  $\mu\text{g}$  and most preferred at least 800  $\mu\text{g}$  per gram chocolate liquor.

Another embodiment relates to a chocolate liquor produced from cocoa beans having a fermentation factor less than 375, the chocolate liquor containing at least 16,500  $\mu\text{g}$  cocoa polyphenols per gram chocolate liquor, advantageously at least 20,000  $\mu\text{g}$ , even better at least 25,000  $\mu\text{g}$  and most preferred at least 30,000  $\mu\text{g}$  cocoa polyphenols per gram chocolate liquor. Preferably, the chocolate liquor contains at least 1,500  $\mu\text{g}$  cocoa polyphenol pentamer per gram

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chocolate liquor, more preferably at least 1,750  $\mu\text{g}$ , advantageously at least 2,000  $\mu\text{g}$ , even better at least 2,500  $\mu\text{g}$  and most preferred at least 3,000  $\mu\text{g}$  per gram chocolate liquor.

Yet another embodiment relates to a chocolate liquor comprising cocoa butter, partially defatted cocoa solids and cocoa polyphenols, wherein the partially defatted cocoa solids contain at least 33,000  $\mu\text{g}$  cocoa polyphenols per gram defatted cocoa solids, advantageously at least 40,000  $\mu\text{g}$ , even better at least 50,000  $\mu\text{g}$  and most preferred at least 60,000  $\mu\text{g}$  cocoa polyphenols per gram defatted cocoa solids. Preferably, the chocolate liquor contains at least 3,000  $\mu\text{g}$  cocoa polyphenol pentamer per gram defatted cocoa solids, preferably at least 3,500  $\mu\text{g}$ , advantageously at least 4,000  $\mu\text{g}$ , even better at least 5,000  $\mu\text{g}$  and most preferred at least 6,000  $\mu\text{g}$  per gram per gram defatted cocoa solids. Preferably, the chocolate liquor is derived substantially from underfermented cocoa beans having a fermentation factor less than 375, advantageously less than 350, even better less than 300 and most preferred less than 250.

##### 2. Partially Defatted Cocoa Solids Having Enhanced Levels of Cocoa Polyphenols

One embodiment of the invention relates to partially defatted cocoa solids having elevated levels of cocoa polyphenols. Preferably, the cocoa solids contain at least 33,000  $\mu\text{g}$  cocoa polyphenols per gram defatted cocoa solids, advantageously at least 40,000  $\mu\text{g}$ , even better at least 50,000  $\mu\text{g}$  and most preferred at least 60,000  $\mu\text{g}$  cocoa polyphenols per gram defatted cocoa solids. Preferably, the cocoa solids contain at least 3,000  $\mu\text{g}$  cocoa polyphenol pentamer per gram defatted cocoa solids, advantageously at least 3,500  $\mu\text{g}$ , even better at least 4,000  $\mu\text{g}$ , more preferably at least 5,000  $\mu\text{g}$ , and most preferred at least 6,000  $\mu\text{g}$  per gram defatted cocoa solids.

Preferably, the partially defatted cocoa solids are derived substantially from underfermented cocoa beans having a fermentation factor less than 375, advantageously less than 350, even better less than 300 and most preferred less than 250.

The partially defatted cocoa solids may be in cake or powder form.

#### D. Methods of Making Novel Edible Products Containing Cocoa Polyphenols

One embodiment of the invention relates to a method of making an edible product containing a cocoa component having an enhanced content of cocoa polyphenols comprising the steps of:

- (a) treating cocoa beans containing a cocoa polyphenols content while conserving a significant amount of the cocoa polyphenols content thereof to form treated cocoa beans;
- (b) producing the cocoa component from the treated cocoa beans; and
- (c) including the component in the edible product.

The cocoa component may be selected from the group consisting of cocoa nib, chocolate liquor, partially or fully defatted cocoa solids, cocoa polyphenol extract and mixtures thereof.

Another embodiment of the invention relates to a method of making an edible product having an enhanced content of cocoa polyphenols comprising adding a cocoa polyphenol additive or a derivative thereof. The cocoa polyphenol additive may be mixed with the other ingredients of the edible composition at any time during the processing or added to the edible product after processing (i.e., spraying cocoa polyphenols onto the product).

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Preferably, the cocoa polyphenol additive is an extract from cocoa beans or a cocoa component thereof. The cocoa polyphenol additive may either be substantially pure (e.g., greater than 95% by weight pure) or mixed with other components.

The cocoa polyphenol additive may either be synthetic or derived naturally.

#### E. Methods of Making Chocolates Having Enhanced Levels of Cocoa Polyphenols

The cocoa components having enhanced levels of cocoa polyphenols may be used to form chocolates by conventional methods.

One aspect of the invention relates to the manipulation of the flavor of the final chocolate product. The use of a cocoa component having higher levels of cocoa polyphenols typically affects the flavor/aroma of the final product. The higher cocoa polyphenols content is typically associated with a bitter, astringent flavor. Various methods may be used to reduce the bitter, astringent note in the cocoa component. According to one embodiment of the invention, flavor additives are used to mask or reduce the flavor/aroma of the product.

This aspect of the invention relates to the use of at least two chocolate liquors having varying levels of cocoa polyphenols. For example, a first chocolate liquor derived from fermented cocoa beans (having a low cocoa polyphenols level) and a second chocolate liquor derived from underfermented beans (having a higher cocoa polyphenols level) are advantageously used. The use of such a blend allows for the production of a chocolate having strong flavor/aroma characteristics as well as enhanced levels of cocoa polyphenols.

One preferred aspect of the invention uses a two step heat treatment (split hot conching) in the processing of the chocolate. The first chocolate liquor having the lower levels of cocoa polyphenols is subjected to a heat treatment at elevated temperatures to develop flavor. Since the first chocolate liquor has lower levels of cocoa polyphenols, it may be subjected to the higher temperature. The heat treated first chocolate liquor is subsequently combined with the second chocolate liquor having the enhanced levels of cocoa polyphenols and further processed into the final chocolate product. Using this method, the chocolate liquor containing the enhanced levels of cocoa polyphenols is not necessarily exposed to the elevated temperatures, thereby preventing a significant reduction in the polyphenols.

One embodiment of the invention relates to a method of making a chocolate comprising the steps of:

- (a) combining chocolate liquor from cocoa beans having a fermentation factor greater than 375 with at least one additive selected from the group consisting of:
  - (i) at least one fat;
  - (ii) at least one sugar;
  - (iii) milk solids; and
  - (iv) mixtures thereof;
 to form an initial mixture;
- (b) heating the initial mixture to a temperature less than about 200° C. for 5 minutes to 24 hours;
- (c) cooling the initial mixture;
- (d) combining the initial mixture with a second chocolate liquor from cocoa beans having a fermentation factor less than 375 and any remaining ingredients to form a secondary mixture; and
- (e) conching the secondary mixture.

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Preferably, the milk solids are in an amount greater than or equal to 12% by weight.

Accordingly, one embodiment of the invention relates to a method of making a chocolate composition comprising the steps of:

- (a) combining a first chocolate liquor from cocoa beans having a fermentation factor greater than 375, cocoa butter and sugar to form an initial mixture;
- (b) heating the initial mixture to a temperature less than about 200° C. for 5 minutes to 24 hours;
- (c) cooling the initial mixture;
- (d) combining the initial mixture with a second chocolate liquor from cocoa beans having a fermentation factor less than 375 and any remaining ingredients to form a secondary mixture; and
- (e) conching the secondary mixture.

Another embodiment of the invention relates to a method comprising the steps of:

- (a) combining a chocolate liquor high in cocoa polyphenols (preferably having a fermentation factor less than 375) with at least one ingredient and heating to a temperature preferably less than 140° C., more preferably less than 100° C. for a period of time between 5 minutes to 24 hours;
- (b) cooling the mixture;
- (c) combining the remaining ingredients; and
- (d) conching the second mixture.

Another embodiment of the invention relates to a method comprising the steps of:

- (a) heating a chocolate liquor high in cocoa polyphenols, preferably a fermentation factor less than 375, to a temperature preferably less than 140° C. for 5 minutes to 24 hours;
- (b) combining the heated chocolate liquor with other chocolate ingredients; and
- (c) conching.

Another embodiment of the invention relates to a method for the production of a chocolate comprising the steps of:

- (a) heating a first chocolate liquor from cocoa beans having a fermentation factor greater than 375 and any remaining ingredients to a temperature less than about 200° C. for 5 minutes to 24 hours;
- (b) cooling the first chocolate liquor;
- (c) combining the cooled first chocolate liquor with a second chocolate liquor from cocoa beans having a fermentation factor less than 375 to produce a secondary mixture; and
- (d) conching the secondary mixture.

Preferably, the fermentation factor of the second chocolate liquor is less than 350, advantageously less than 300, even better less than 275, and most preferred less than 250.

According to a preferred embodiment, the fermentation factor of the second chocolate liquor is less than 225, advantageously less than 200, even better less than 150, and most preferred less than 125.

#### F. Methods of Producing Cocoa Butter and Partially Defatted Cocoa Solids

Yet another aspect of the invention relates to the production of cocoa butter without necessarily the concomitant conservation of polyphenols. This aspect of the invention relates to a method for processing cocoa beans to make cocoa butter and cocoa powder. In particular, the method comprises the steps of cleaning and preparing the cocoa